The Contaminant Analysis Automation Project

LALP-95-110
April 1995

This publication was
produced by the
**Communication Arts & Services Group
(CIC-1)**
Writing and Editing
Ann Mauzy
Graphic Design
Zizi Kolshorn
Printing Coordination
Guadalupe Archuleta

Los Alamos
NATIONAL LABORATORY
Los Alamos, New Mexico 87545

A U.S. DEPARTMENT OF ENERGY LABORATORY

CONTENTS

AN OVERVIEW OF THE CONTAMINANT ANALYSIS AUTOMATION (CAA) PROGRAM

by

Matthew Monagle, Environmental Management Division,
Los Alamos National Laboratory, Los Alamos, NM,
Frank Settle, Consultant, Lexington, VA, and
Leon Klatt, Oak Ridge National Laboratory, Oak Ridge, TN

INTRODUCTION

Mission

The mission of the CAA Program is to improve methods for chemical analysis of environmental samples. To accomplish this mission, the CAA team is developing automated laboratory systems based on a plug-and-play strategy for integrating components. Realizing that standardization is the key to implementing this strategy; CAA will develop, demonstrate, and encourage commercialization of standards for laboratory automation. While the CAA mission is driven by the analyses in support of the extensive remediation programs of the Departments of Energy and Defense, it impacts any industry that depends upon high volumes of repetitive chemical analysis.

Benefits

Laboratory automation, including effective data management, can improve chemical analysis by reducing analysis cost per sample, increasing precision and accuracy of results, minimizing exposure of personnel to hazardous materials, and decreasing the time between sample submission and production of the final report. Reduced cost per analysis results from increased sample throughput and continuous operation with minimal operator intervention.

Applications

The CAA program focuses on the most common methods required by DOE and DoD for environmental remediation. Studies have shown that approximately 80% of the environmental analyses for these departments can be meet by nine analytical methods. Flexible automated systems using plug-and-play concepts can meet this need. The CAA program is working closely with regulatory agencies to validate automated methods and to assure regulatory acceptance of results obtained with automated systems.

THE CAA PARADIGM

Basic Components

Every analytical method contains three basic operations: sample preparation; measurement; and data interpretation (Figure 1). The measurement component has received the most attention to date, with many vendors providing sophisticated, automated instrumentation. The initial component, sample preparation, remains time consuming, labor intensive, potentially hazardous to personnel, and a major source of errors. Currently, the final component, data interpretation (including quality assurance and report generation), consumes about 50% of the staff resources in environmental laboratories. Furthermore, the data interpretation tasks require the expertise of an experienced analyst. Thus, the first and last components present the greatest opportunities for the CAA program to improve automated methods.

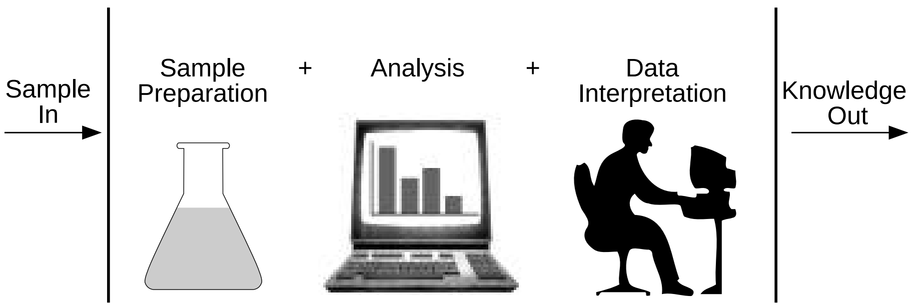

Figure 1. Basic operations of analytical methods.

System Integration

In addition to focusing on the automation of sample preparation and data interpretation, the CAA Program addresses the greater challenge of combining these components with measurement instruments to form totally integrated systems. At present, many individual system components have been automated by vendors of instruments and equipment, but the integration required for a totally automated, multi-vendor system remains a difficult task. Lack of integration standards requires each system to be custom built by either the vendor, the customer, or a third-party system integrator.

Hardware and Software

In order to achieve its goal of developing automated laboratory systems, the CAA Program is developing and demonstrating an implementation of hardware and software standards for system integration. Laboratory automation should be analogous to office automation, where standard hardware interfaces and software drivers facilitate rapid integration of computers, printers, modems, CD ROMs, and other components into efficient, automated systems.

Plug-and-Play Concept

The plug-and-play concept overcomes problems that have hindered automation of environmental analysis. Diverse matrices, such as soil and water, will not require separate automated systems. Rather, the replacement of the sonication component (for extracting analytes from soil samples) with a liquid-liquid extraction component (for removing analytes from aqueous samples) allows the same system to process both sample matrices. Similarly, the introduction of a procedure, such as Florisil™ cleanup of soil samples contaminated with polychlorinated biphenyls (PCBs), can be accomplished by plugging a Florsil™ column component into the automated system. The system's master controller identifies the new component, reconfigures the system, and manages the flow of samples through all components.

The Standard Laboratory Module™ (SLM)™

The basic component of the CAA system is an SLM.[1] Each SLM performs a group of laboratory unit operations (LUOs) required by an analytical procedure. An embedded microprocessor on the SLM is programmed to direct the execution of LUOs. For example, a Soxhlet SLM, used to extract analytes from solid samples, contains liquid-handling and -heating LUOs that are controlled by the SLM's embedded microprocessor. SLMs communicate with the system controller, known as the master controller or task sequence controller (TSC), through a standard hardware interface, which can be either serial (RS232 or Ethernet) or parallel (GPIB or IEEE-488).

The software interface consists of sets of standard commands and events that are recognized by each SLM and the master controller. The internal operations of individual SLMs are independent of these standard commands and events. However, each SLM must respond to standard commands and queries from the master controller with a standard response from a set of standard events. Commands and queries that are not applicable to a particular SLM must be handled in a uniform manner as defined in the SLM Software Specifications.[2] Once the sample is delivered to an SLM in the ready state, and processing is initiated, the SLM requires no further interaction with any system component, including the master controller, to process the sample. When processing is complete, the SLM notifies the master controller. If the SLM malfunctions while processing a sample, it must be able to inform the master controller without being queried. The SLM must be able to respond to queries from the master controller at all times.

Variations of SLMs that can be interfaced to the master controller include: standard service modules (SSMs), analytical instrument modules (AIMs), and data interpretation modules (DIMs). SLMs handle tasks associated with sample preparation (sonication, extraction, filtering, concentration, separation, and dissolution), AIMs perform measurements, DIMs interpret data, and SSMs transfer samples (robots for containers; pumps for fluids). Robot-SSMs can enter the space of SLMs or AIMs in order to transfer samples and consumables. However, an SLM may not interact directly with another SLM or AIM.

Sample transfers among SLMs and AIMs are handled by SSMs under the direction of the master controller. Although, AIMs have embedded computers and the required hardware ports to interface with the master controller, software modifications are required for the AIMs to interact with the master controller using the standard sets of commands and events. The DIM is a software module that interprets data from an AIMs using a combination of chemometric methods. Each analytical method has a unique DIM.

Additional Components

In addition to the master controller and modules described previously, two other components are required to complete a system: a human computer interface (HCI) and a dedicated database. The HCI is used by the operator to enter and track samples, monitor the system, maintain the modules, and access the database. The database contains information on analytical methods, maintenance procedures, module capabilities, samples and sample processing, raw and processed data, operator qualifications, and final results. Audit trails and processing diaries for each sample are also stored in the database. The database can transfer information to a laboratory information system (LIMS) which then generates reports required by customers.

Standard Analysis Methods (SAMs)

Fully automated systems are capable of performing standard methods of analysis. The configuration of components and directions for each procedure required by the method are stored in the database. When the operator uses the HCI to select a SAM and properly configures the system, the master controller checks the system configuration and retrieves the information required to perform the SAM from the database. The configuration of the SAM for PCBs in soils is shown in Figure 2.

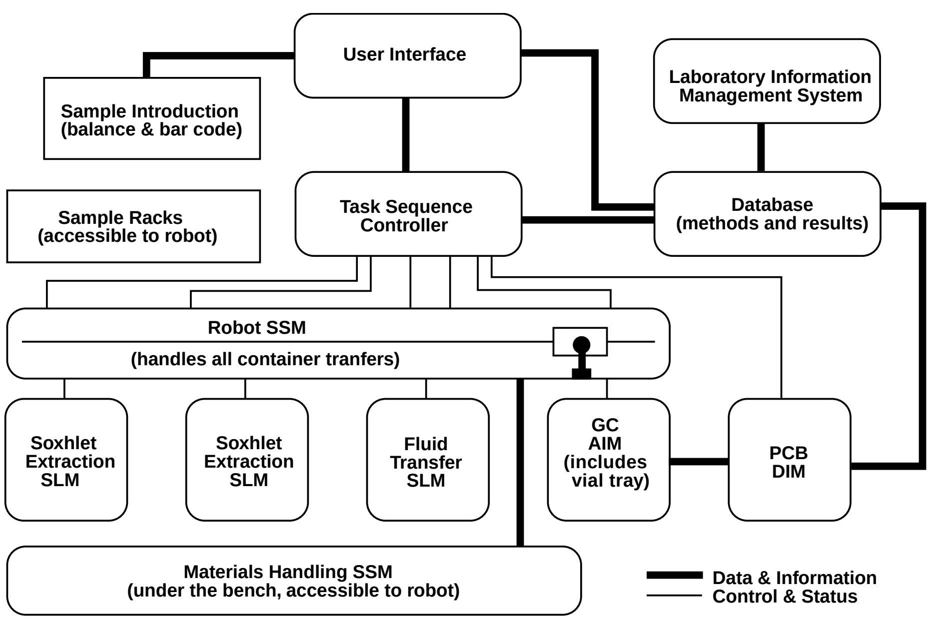

Figure 2. Configuration of the SAM for PCBs in soils.

THE PATH TO FULL AUTOMATION

Near-Term Concerns

The CAA team realizes that in the near term many laboratories may not be able to justify the cost of a totally automated system regardless of its flexibility. With this in mind, a path to fully automated systems exists that allows the purchase of individual SLMs to operate in a standalone mode. Such SLMs can alleviate bottlenecks in sample processing; their embedded microprocessors, keypads, and displays permit direct interaction with operators. Since SLMs are built to standard specifications, they have the capability to become components of larger more automated systems.

The Mini-SAM

A mini-SAM represents an intermediate mode of integration between the standalone SLMs and fully automated systems that can perform all tasks associated with a SAM. The mini-SAM is a group of two or more SLMs that perform several procedures required by a method. The mini-SAM shown in Figure 3, extracts the analytes from a sample by sonication, rapidly concentrates the resulting solution, and transfers it to a gel permeation chromatograph for cleanup before gas chromatographic analysis. Samples can be transferred among these SLMs by a technician or a robot. This combination of SLMs eliminates a common bottleneck in many organic methods. Addition of sample entry SSM, HCI, database, AIM, and DIM are required for this mini-SAM to become a fully automated system.

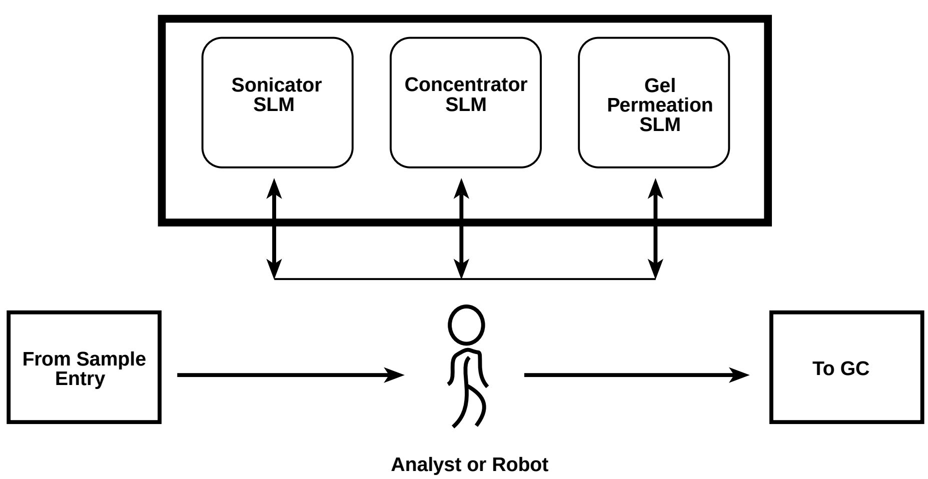

Figure 3. The mini-SAM.

ATTRIBUTES OF SLMs AND THE INTEGRATED SYSTEM

Layout

There are no specific dimensions for an SLM. Most SLMs should be designed to operate in a standard laboratory hood and should be compatible with this environment. Since there will be several SLMs in a fully integrated system, each SLM should be as small as possible without hindering routine maintenance and repair work. All interface connections (including those for utilities, gases, and liquids) should be located on the front or rear panels. However the distance between interface connections must facilitate connect / disconnect operations. Sample container input and output ports must be located on the front panels and accessible to a robot.

Compressed gases common to laboratories (nitrogen, helium, and filtered, dehumidified, compressed air) will be supplied from sources external to SLMs. In cases where an SLM requires an unusual gas such as P10 (10% methane in argon), the required connection should be provided by the SLM vendor. Because many laboratories do not provide common vacuum lines, it is recommended that SLMs requiring vacuum contain their own vacuum systems.

Liquid solvents for a fully integrated system will be supplied from a common reservoir or from a reservoir built into the SLM. The reservoir should consist of a tank lined with inert material with inert gas pressure applied to transfer the solvent.

Sample Transfer

All SLMs should be able to transfer samples to other SLMs as required to perform the specified method. There are two ways to transfer samples, container transfer and tube transfer. The robotic SSM performs container transfers, and the fluid SSM handles tube transfers. The robot module handles vials and beakers of varying sizes and configurations. The tube transfer module allows direct routing of a sample to SLMs without the capacity to retrieve the sample from a beaker or vial. Because of the different sample sizes encountered in an integrated system, it's not possible to have rigid standards for container sizes or tube transfers.

Container transfer is relatively simple and can take advantage of queuing. However, differences in container sizes can cause problems with robotic manipulators. One solution is to use SLM inputs and outputs that accommodate different-sized containers. For example, the CAA high-volume concentrator SLM input lowers a tube into the sample input container to transfer the sample into the SLM and outputs the concentrated sample to a smaller vial for gas chromatography.

Each SLM input and output port must be accessible to a robot for container transfer. Inputs or outputs that require threading or snapping containers into place are not acceptable. Alignment of narrow-mouth sample containers in ports is critical. Grooves or rails will be used to assist in positioning these containers. Several current SLMs use pneumatic cylinders to position the sample container or to lower a tube into the container to transfer sample into the SLM.

Tube transfer eliminates the requirement for robotics interaction and the problems associated with sample containers. However, this transfer method also has potential problems. Extensive rinsing of all tubing is required to avoid cross-contamination, and extra pumping hardware must be designed into the system. The use of tube transfer eliminates the possibility of queuing; if a fully integrated system uses tube transfer and requires queuing, sample transfer must be supplemented by a robot.

Capping

Samples outside SLMs should be covered with a suitable lid to minimize introduction of material that might contaminate the sample. Removal of lids from samples is not an SLM function. This task will be completed by the robotics system or the operator before entering samples into SLMs. In cases where samples from an SLM (1) will sit open for any length of time, (2) have a low solvent volume, or (3) have an accurately measured volume, provision has been made for applying a sealing cap and piercing a capped container. The cap material should be carefully selected to minimize contamination.

Material Flow

Materials such as samples, glassware, solvents, and wastes are transferred to and from individual SLMs by the operator, a robot, or a tube transfer device. Once an SLM has all the materials required for a specified procedure, the master controller initiates the procedure. CAA standards require all SLMs to perform their LUOs independent of all other SLMs; thus the robot is used for handling materials outside the boundaries of the SLM.

SLM Ports

Each SLM should have enough port capacity to handle the maximum number of samples it can process simultaneously. For example the high-volume concentrator can process only one sample at a time, the Soxtec, two samples, and the GPC up to six. Excess sample container capacity should reside in queues outside the boundaries of the SLMs. The ports are too valuable to use as sample queues. SLM ports are locked (available for sample transfer) while samples are being processed or unlocked (unavailable for sample transfer) when the SLM is not processing samples. A port that can receive a sample is in the unlocked state. When the master controller determines that a port is unlocked, it locks the port, transfers the container, checks to see if the container was accepted, and, if it was, unlocks the port.

Each SLM has a single input port for all solvents; for procedures requiring the delivery of multiple solvents to a single SLM, the system must be able to switch the appropriate solvent to the SLM solvent input port. The system should also be able to rinse lines leading to the common input port. For standalone operation, the SLM vendor should supply solvent containers. Surrogates and spikes should be added to samples by either the operator at the time of sample entry or a separate SLM dedicated to this function.

Queues and Queuing Interchanges

Queues do not exist within individual SLMs. Queues have two functions in automated analytical systems. First, they can hold a few samples that are awaiting processing because of differences in processing times of component SLMs. These queues are not intended to improve throughput but rather to insure smooth processing of the samples through the system. The second function of a queue is to improve overall laboratory operations. This type of queue can be accomplished in several ways depending on the laboratory environment. Samples may undergo preparative and separation procedures using appropriate SLMs in the automated system but may then be measured with an instrument outside the system. A queue becomes a holding place for these samples. If there is no SLM for a given procedure, a queue is required for samples awaiting manual processing. This queuing function allows integration of an automated system with instruments and manual procedures outside the system.

Embedded SLM Controllers

In order for an SLM to perform its tasks independent of an external master controller, an SLM must have embedded intelligence. This on-board intelligence allows the master controller to send the SLM a standard command and associated parameters to initiate a procedure. Once the SLM starts the procedure, it retains control of its operations. The embedded SLM controller is responsible for directing all tasks (LUOs) associated with the procedure, monitoring the process, and informing the master controller when the sample processing is complete. When the embedded controller detects an error, it informs the master controller immediately. This distributed intelligence minimizes master controller-SLM interactions and reduces the system's information processing needs.

Hardware Communication Interface

SLMs communicate with the system controller (master controller) through RS-232 or Ethernet serial ports, or GPIB or IEEE-488 parallel ports.

Software Interface

This software is the key to implementing CAA's plug-and-play strategy. The interface is defined in terms of SLM capabilities, data sets, and message exchanges between SLMs and the master controller. The message content is independent of communications hardware. Once the master controller determines that an SLM can perform a procedure, the master controller initiates the procedure. The master controller is not concerned with how an SLM performs the procedures, only that the SLM can complete the procedure with the given operating parameters.

A standardized state diagram is used to describe the interactions of the master controller with SLMs and other system components. These states describe communications, remote and local control of SLMs, SLM processes, and alarms. A set of standard commands and queries for communication between the master controller and SLMs has been defined. SLMs respond to master controller commands and queries through a standard set of events. The commands, queries, and events are primary elements of the software drivers for the master controller and SLMs. Each SLM has a capabilities data set that describes its functions and operational limits to the master controller.

Data Flow

The flow of data and information through the automated system is as important as the flow of samples through the system. Initially the database sends method procedures and parameter values to the master controller. As a sample moves through the system, each SLM sends processing information through the master controller to the database. The instrument (AIM) sensing analyte concentrations sends measurement data through the master controller to the database. This raw data is requested by the DIM, where it is converted into information, and this information is stored in the database. All the information for a given sample generated by the SAM is transferred to the LIMS where it is integrated with other information on the sample, and a final report produced. The flow of data and information conforms to approved validation and archiving procedures such as EPA's Good Automated Laboratory Procedures and ISO 9000 protocols.

Since environmental analyses are highly regulated, it is important to capture all pertinent information. In addition to raw data from the measuring instrument and the final analytical results, time stamps indicating when each sample was processed by the system SLMs', other information on processing, and all processing errors are recorded.

Standalone Operation

SLMs should be able to operate as independent units as well as components of automated systems. This allows laboratories to alleviate bottlenecks in analytical methods and also to follow the path to fully integrated systems described previously. Standalone SLM operation supports the same features that are supported when the SLM is functioning as a component of an automated system.

The SLM / operator interface is a menu-driven keypad and display. Two modes of standalone operation are available; one for maintenance and the other for processing samples. When problems are encountered in sample processing, a standalone SLM should be able to notify the operator via the SLM's display. The intelligence programmed into the SLM's onboard microprocessor is the key to implementation of these features.

In standalone operations, the human assumes the functions of the integrated system's master controller and the container handling system. The operator thus provides values for all method parameters, containers, and consumables, and also inputs all samples. Once the processing of samples is initiated, the operator monitors the process and responds to any errors. When the processing is complete, the operator removes the samples and wastes.

Validation of the Components and the Integrated System

Analytical methods, procedures, and equipment must be validated to be accepted by the regulatory and analytical communities. Validation demonstrates that the specified analytical methodology can deliver precise, accurate results reliably.

Each SLM must be validated for each intended application. For example, an SLM developed for PCB and semivolatile organic extractions must be validated separately for each extraction procedure. This approach is consistent with the CAA's plug-and-play approach to integrated systems. It (1) provides the end user with information on the reliability of the SLM, (2) establishes the analytical performance of the SLM for specified procedures by measuring surrogate and matrix spike recoveries, and (3) determines the ability of the procedure to handle a diverse range of samples. Validation of an individual SLM reduces the need to revalidate the full integrated system when a new SLM is introduced. A limited system validation in conjunction with ongoing quality control should be sufficient to prove that an integrated system is validated.

The CAA program has developed standard procedures for validating each system component. These procedures describe the use of a data requirements documents to define the validation process. This document addresses issues affecting the validation such as the purpose of the validation, the end users of validation data, and the expected outcomes of the validation. Parameters to be measured and the documentation required for validation are also included in the standard procedure. For example, an extraction SLM should be evaluated on the basis of the following parameters: blank samples, detection limits, cross-contamination, QC sample / standard-reference-material sample results, and actual sample data.

CURRENT STATUS AND FUTURE DIRECTIONS

The CAA program has designed and constructed prototypes of the components (SLMs, master controller, HCI, DIM, and database) required to demonstrate an integrated automated system for the analysis of polychlorinated biphenyls and semivolatile organic compounds by gas chromatography. Standardization of hardware and software interfaces between system components and the master controller has allowed the plug-and-play concept to be used in the implementation of this system. Single laboratory validation of several SLMs has been completed. Draft documents describing the SLM software interface and common specifications for SLMs have been prepared. Appendix 1 shows the status of all CAA SLMs and other system components. The CAA program is finalizing an agreement with SciBus Analytical, Inc., a system integrator, to transfer this technology to the commercial sector.

Much work remains before the environmental analysis needs of the Departments of Energy and Defense can be met with cost-effective automation systems. The CAA Program is defining SLMs and SAMs for metals and radiochemical analyses. The conversion of textual analytical procedures into computer-readable scripts that allow the master controller to direct SAMs is an important feature to be developed. While the initial focus of the CAA Program has been on environmental analysis, potential applications for integrated, automated analysis systems extend into many areas including pharmaceuticals, chemicals, foods, and clinical analysis. Successful implementation of the plug-and-play concept for implementing integrated, automated systems can have a major impact on high-volume chemical analysis.

[1] Salit, M. L., Guenther, F. R., Kramer, G. W., Griesmeyer, J. M., *Anal. Chem* **66**(6), 1994, 361A — 367A.

[2] CAA SLM Software Specifications are available upon request.

APPENDIX A. CAA SYSTEM COMPONENTS

1. Sample Preparation Modules

SLM	Methods	Developer(s)	Status
drying column	EPA 3541 & 3550A	INEL	prototype completed
high-volume concentration	all organic methods	INEL	completed & tested
hot plate	EPA 3550, 3010, 3020	PNL & Zymark	prototype completed
digestion filtration	EPA 3541 & 3550A	INEL & ORNL	prototypes completed
sample capping	EPA (acid digestion)	PNL	prototype completed
microwave digestion	EPA 3051, 3015	ORNL & CEM	prototype completed
soil preparation	grinds soils into fine particles	U. Fla.	under development

2. Analyte Separation Modules

SLM	Method	Developer(s)	Status
Soxhlet extraction	EPA 3541	PNL & Tecator ABC Instruments	completed & tested
sonication extraction	EPA 3550A	LANL/INEL	completed & tested
column adsorption (solid phase extraction)	EPA 3610, 3611, & 3620	PNL	under development
ion exchange column	EPA 9080	ORNL	under development
gas chromatography (column)	EPA 8080	LANL & Varian	under development completed
gel permeation chromatography (GPC)	EPA 3640A	INEL & ABC Labs Instruments	completed & tested
liquid-liquid extraction	EPA 3510 & 3520	PNL & ABC Labs Instruments	under design
coprecipitation	LANL ER 110	ORNL	under design

3. Measurement Modules

AIM	Method(s)	Developer(s)	Status
gas chromatography (detector)	EPA 8080A	LANL & Varian	completed
GC-MS	EPA 8250A	LANL	under design
atomic absorption	EPA (analyte specific)	ORNL	under design
IPC-AES	EPA (analyte specific)	ORNL	under design
laser ablation MS	screening	ARCO Power Technologies	under development
alpha counting	EPA & DOE (analyte specific)	ORNL	under design

4. Data Interpretation Modules (DIMs)

DIM	Method(s)	Developer(s)	Status
GC PCB (Acrolor)	EPA 8080A	LANL, U. Tenn., Fla. State U., Varian, Thru-Put Systems, and Neural Ware	under development
GC-MS (volatile organic compounds)	IPA 8250A	LANL	under development

5. Standard Support Modules (SSMs)

SSM	Method(s)	Developer(s)	Status
sample introduction	introduction of samples into system by operator	U. Fla.	under development
robot	using containers for sample transfer	ORNL & Hewlett Packard	completed
fluid transfer	using tubes for sample transfer	INEL	completed
materials handling	requiring sample containers	U. Texas	completed
solvent recovery	all using organic solvents	INEL/ABC Labs	completed
filtration	all methods requiring filtration	INEL	completed

6. System Control and Information Management

Component	Function	Developer(s)	Status
task sequence controller (TSC)	system control	SNL	under development
human-computer interface (HCI)	operator-system interaction	LANL & ORNL	under development
database	storage of information, data, and results	LANL	under development
training & maintenance info system	maintenance & instructions for operation	U. Fla.	under development
bench manager	intelligent management of samples, blanks, & standards for EPA compliance	SNL	predesign phase
laboratory information	interface between CAA management database and LIMS	LANL & LIMS vendor	under design

APPENDIX B.THE CAA TEAM

System Integrator

SciBus Analytical, Inc.

DOE Laboratories

INEL	Idaho National Engineering Laboratory
LANL	Los Alamos National Laboratory
ORNL	Oak Ridge National Laboratory
PNL	Pacific Northwest Laboratories
SNL	Sandia National Laboratories

Universities

University of Florida, Gainesville, Mechanical Engineering and Nuclear Engineering Sciences Departments
University of Tennessee, Knoxville, Electrical and Computer Engineering Departments
University of Texas, Austin, Mechanical Engineering Department and Robotics Research

Indistrial Partners

ABC Instruments, Inc., Columbia Missouri
ARCO Power Technologies, Inc., Washington, DC
CEM Corp., Matthews, North Carolina
Environmental Science and Engineering, Inc., Gainesville, Florida
Hewlett Packard Corp., Palo Alto, California
Neural Ware, Inc., Pittsburg, Pennsylvania
Thru-Put Systems, Inc., Orlando, Florida
Varian Chromatography, Palo Alto, California
Zymark Corporation, Hopkinton, Massachusettes